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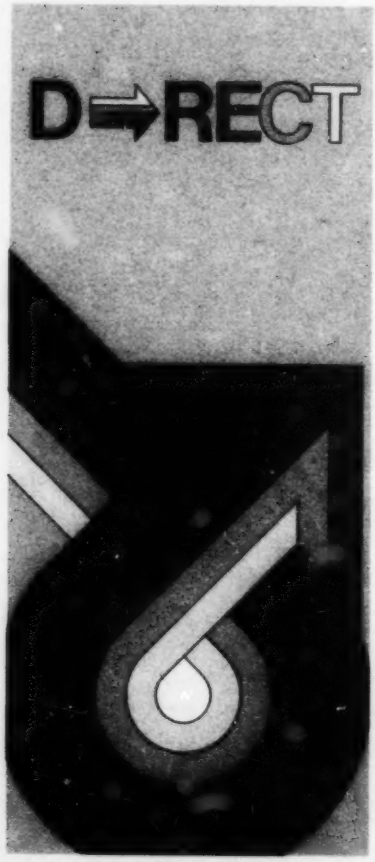

Construction and Demonstration of a Portable Chemical Processing Unit for Purifying and Recovering Waste Gas Treating Chemicals from the Petroleum Industry

Report EPS 3/PN/1
September 1992

Canada

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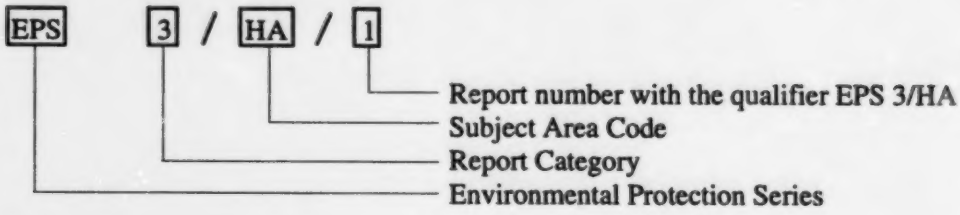

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Construction and Demonstration of a Portable Chemical Processing Unit for Purifying and Recovering Waste Gas Treating Chemicals from the Petroleum Industry

by

T. Beasley and D. Merritt
Canadian Chemical Reclaiming Ltd.

for the

Technology Development Branch
Environmental Protection
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This project was conducted under the Development and Demonstration of Resource and Energy Conservation Technology (DRECT) Program

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Abstract

Gas processors are frequently faced with problems associated with accumulated impurities in gas-treating chemicals which are used to remove water vapour and acid gases such as hydrogen sulphide or carbon dioxide. These impurities come from materials present in the gas being treated and from degradation products which are produced by chemical reactions in the treating chemical as it is circulated.

After researching various alternatives, a new process has been developed for purification of gas-treating chemicals employing high vacuum distillation under conditions of flow and temperature such that no risk of further degradation is allowed. This process effectively purifies the treatment chemicals to such a level that they can be used as makeup or fresh chemical in the gas-treating process.

This process uses a small amount of natural gas as fuel to effect the distillation. Energy savings are realized, however, through reduced production requirements for virgin gas treatment chemicals and reduced requirements for incineration of the spent chemicals. The result is substantial energy savings.

Résumé

Les sociétés qui s'occupent de traiter le gaz naturel font souvent face à des problèmes liés à l'accumulation d'impuretés dans les produits chimiques utilisés pour débarrasser le gaz de la vapeur d'eau et des gaz acides qu'il contient, comme l'hydrogène sulfuré ou le dioxyde de carbone. Des substances présentes dans le gaz pendant le traitement ou des produits de dégradation résultant de réactions chimiques dans le produit de traitement en circulation sont à l'origine de ces impuretés.

Après avoir cherché diverses solutions de rechange, nous avons mis au point un nouveau procédé permettant de purifier les produits chimiques utilisés pour traiter le gaz naturel; ce procédé fait appel à la distillation sous vide poussé dans des conditions de débit et de température éliminant tout risque de dégradation ultérieure. Ce procédé permet de purifier efficacement les produits de traitement, au point qu'on peut ensuite les réutiliser, dans le procédé de traitement, comme produits d'appoint ou encore comme produits vierges.

Dans ce procédé, une petite quantité de gaz naturel est consommée pour produire la chaleur nécessaire à la distillation. On réalise néanmoins des économies d'énergie en raison de la fabrication de quantités moindres de produits chimiques de traitement et de l'incinération de quantités réduites de produits chimiques usés. Les économies d'énergie ainsi réalisées sont appréciables.

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The cooperation and assistance extended by the DRECT program manager during the execution of this contract are also gratefully acknowledged.

Section 1

Introduction

The operator of a gas processing plant will normally use glycol chemical solutions for the dehydration of produced natural gas and amine chemical solutions for removal of acid gases, such as carbon dioxide (CO_2) and hydrogen sulphide (H_2S). The chemical solutions frequently accumulate undesirable contaminants over time which interfere with the ability of the solution to perform as effectively as required.

When contaminants have accumulated to the point that the chemical solution is no longer capable of performing adequately, and remedial measures such as fresh chemical addition or caustic addition have been exhausted, the operator is faced with removing the waste solution from the plant, replacing it with virgin chemicals and disposing of the exhausted solution.

The waste solutions are classified as hazardous, and must be disposed of in an appropriate manner. In Alberta, this means transportation and disposal at Swan Hills.

The principle objective of this project was to develop and demonstrate a new technology that offers the petroleum industry an opportunity to economically regenerate their waste gas treating chemicals, reduce the total waste volume, and save energy required to manufacture and dispose of process chemicals.

Commercial feasibility of the proposed technology was considered sound with the achievement of the following objectives:

1) Demonstration of 90 to 95% recovery of three spent gas treating chemicals (i.e., one glycol, one amine, and one sulfinol or sulfolane).

2) Demonstration that 90 to 95% of the contaminants identified in the spent gas treating chemicals are removed from the reclaimed products.

3) Demonstration that the reclaimed products are marketable.

4) Demonstration that the waste byproducts of the reclaiming process can be contained and disposed of in an environmentally acceptable manner.

5) Confirmation that the new technology can be employed as a service to the petroleum industry and result in economic, environmental, and energy benefits.

Section 2

The Gas Processing and Treatment Industry

Most of the Canadian natural gas production and processing industry is located in Alberta. At present, this industry is experiencing a healthy rate of growth stemming from the free trade agreement recently signed between Canada and the United States.

In addition to the hydrocarbon content (principally methane) certain constituents are commonly found in natural gas as it is produced from the well. Water vapour and various combined forms of sulphur, particularly hydrogen sulphide, are present and these are unacceptable contaminants in gas sold to the public.

Water vapour will form solid gas hydrates (complex physical combination of water and hydrocarbons) at reduced temperature and pressure. Water vapour must, therefore, be removed from the consumer gas supply (sales gas) to avoid deposition of hydrates in piping and equipment.

Acid gases are corrosive and sulphur compounds are toxic as well. These constituents must also be removed from sales gas to meet safety and corrosion criteria.

2.1 Gas Treatment Processes

The removal of water vapour from natural gas is accomplished in dehydration units where a physical absorbent (a solution of one of the ethylene glycol family) is used to absorb the water vapour from the gas by contact in a column with vapour-liquid contact trays. This is not a chemical reaction but rather a physical dissolving of the water vapour in the glycol. The glycol with dissolved water is regenerated by steam stripping. The water vapour is removed and the regenerated glycol is recirculated to the absorber.

The removal of the acid gas constituents from the natural gas is accomplished in "amine" treating units in which a solution of one of the amine family of chemicals is contacted with the natural gas in a column with vapour-liquid contact trays. The amine, which may be any of a group of chemicals, described in the following section, is a weak base and reacts with the acid gas in the contactor. This chemical combination of the sulphur (usually in the form of hydrogen sulphide) or carbon dioxide with the amine can be regenerated using steam stripping. With regeneration, the acid gas is removed from the amine solution in the regenerator and the amine is recirculated to the absorber.

2.2 Gas Treatment Chemicals

The chemicals which are commonly circulated in gas processing plants include ethylene glycol (EG), diethylene glycol (DEG), triethylene glycol (TEG), monoethanolamine (MEA), diethanolamine (DEA), sulfinol, and methyl diethanolamine (MDEA). Each of these chemicals has its own group of degradation products and, in addition, each source of gas being treated may have different contaminants which can be left behind in the chemical solution.

Each gas treating chemical is temperature sensitive, and has a temperature limit, above which decomposition will begin. The temperature limits vary but are all in the general region of 205^{o}C.

Monoethanolamine has a low atmospheric boiling point that permits the operation of traditional in-plant reclaimers at slightly more than atmospheric pressure without danger of reaching the lower temperature limit. As a result, this solvent does not normally present

the same disposal problem as other, higher boiling, chemicals.

All of the other gas treating chemicals have high boiling points, (i.e., DEA, DEG, TEG, MDEA, sulfinol). They are subject to decomposition if distilled in a reclaimer under atmospheric pressure. Some in-plant reclaimers have been designed and installed for these chemicals but, in general, have not been successful in operation and have been abandoned.

It is difficult to identify the numerous degradation products which result from the partial decomposition of treating chemicals. Amine products, in general, result from a reaction between the chemical and carbon dioxide (CO_2) to form various families of oxazolidones, piperazines and other undefined families of nitrogen-based compounds which are usually chemically inactive (Polderman and Steele, 1956; Kennard and Meisen, 1980; Kim, 1988).

It is easier to recognize the impurities that are brought into the gas-treating chemical by the gas or by the reaction between the gas and the steel piping and equipment in the plant. These materials include salt, asphaltenes, sand, oil and grease, suspended solids, and fine particles of iron sulphide. In addition, the pH of the circulating solution may be affected.

When thiosulphates or cyanides are present, a reaction takes place to form amine thiosulphates or amine thiocyanates. These materials are called heat stable salts and, unlike the pure amine cannot be regenerated by heat and steam stripping. Although these materials do not absorb acid gas, they remove some of the active amine from solution. Addition of caustic soda or soda ash will "free" the combined amine but will result in increased total dissolved chemical in the circulating solution, adding to viscosity and impeding absorption.

As the level of impurities in the chemical solution increases, the operator may experience difficulties with foaming, corrosion, or loss of capacity to remove water or acid gas.

Foaming is promoted by the presence of oil and grease and of finely divided iron sulphide. Usually the foaming can be controlled by the addition of anti-foam agents such as silicones. While this may become expensive, it will not ordinarily require the shutdown of the plant to remove the chemical solution.

A suspected source of corrosion in a gas plant is the amine family of degradation products. Although authorities are not in agreement on this subject (Schmeal *et al.*, 1978; Blanc *et al.*, 1982; Chakma and Meisen, 1986) it is evident that the likelihood of corrosion increases as the concentration of degradation products increases. A second and more certain source of corrosion is stress corrosion cracking. When a significant salt (sodium chloride, NaCl) content is present in systems that contain austenitic stainless steel stress cracking occurs. Significant salt concentrations, above 200 to 400 ppm, may require plant shutdown and the replacement of chemical to avoid serious failure due to stress corrosion cracking.

A combination of loss of absorption capacity and an increase in viscosity ultimately reduces the overall capacity of the gas treating solution. At this time, more pure chemical would have to be added to the solution and/or the circulation rate would have to be increased. When the viscosity is already increased because of contaminants, however, more pure chemical will further increase the viscosity and at the same time reduce the pumping capacity of the process. This may result in a vicious circle in which the increasing loss of capacity of the plant cannot be prevented. If this gets to a point where the plant cannot do its job, the chemical charge must be disposed of, the plant flushed, and new solution must be added.

Section 3

Disposal Alternatives

Increasing public awareness of potentially hazardous wastes has decreased the options for disposal of spent gas treating chemicals. Federal and provincial laws concerning waste management and disposal, have made it more difficult and more costly to dispose of waste gas treating chemical solutions.

In the past it is likely that waste chemicals were dumped in remote areas or in landfill sites; pumped down disposal wells or lagooned and left to degrade naturally. An example of a newer and much more costly alternative is the Swan Hills Hazardous Waste Disposal facility in Alberta.

One reclaiming system that has been in use for some time uses activated carbon to remove impurities from the gas-treating chemical. This system can be effective but is quite expensive.

Canadian Chemical Reclaiming Ltd. initiated a study to try to solve the disposal problem. The range of contaminants that treating chemicals contain was studied and various purification systems were investigated.

Working models of process schemes were constructed and a laboratory was set up for testing and evaluation.

The objective of the work was to develop a process that could economically remove high percentages of the contaminants identified.

The portable plant in operation today is the result of this development work. The plant incorporates a vacuum distillation system to remove impurities without subjecting the gas-treating chemical to the high temperatures that cause decomposition. This innovative design is in patent-pending status. The unit is portable so that the waste chemical may be processed at the site of the gas plant.

The portable plant reduces the volume of waste to a fraction of the original contaminated chemical solution; 90 to 97% of the chemical solution can be returned to the gas plant for reintroduction to the gas-treating process.

Section 4

Potential Energy Savings by Remanufacture

A preliminary market survey of waste gas treating chemicals indicates a minimum of 1 818 436 L (400 000 gal) of these materials will require reprocessing each year. The portable reclaiming unit can process this quantity of waste chemicals while operating an average of only eight hours per day and five days per week. As the unit has been designed to operate continuously twenty-four hours per day, seven days per week; it has the capacity to remanufacture a much larger quantity of waste chemicals.

Based on the figure of 1 818 436 L/yr (400 000 gal/yr), the potential energy savings can be estimated. To prepare this estimate, it has been assumed that only ethylene glycol is being processed. The estimates using ethylene glycol are:

1) Energy to manufacture (Union Carbide, Dow)... 16 282 kJ/kg (7 000 Btu/lb)

2) Energy to Destroy (Swan Hills Waste Disp.)... 46 520 kJ/kg (20 000 Btu/lb)

3) Energy to Remanufacture (Can. Chem. Reclaim)... 2 791 kJ/kg (1 200 Btu/lb)

Two basic disposal possibilities are considered:

a) Send the waste chemical to Swan Hills and purchase replacement glycol from the manufacturer.

Energy = 16 282 kJ/kg (purchase) +
46 520 kJ/kg (Swan)

= 62 802 kJ/kg (27 000 Btu/lb)
to dispose and replace

b) Remanufacture the waste chemicals with losses of no more than 10% of the feed material. These losses must be sent to Swan Hills and also replaced through purchase from the manufacturer.

Energy = 2 791 kJ/kg (remanuf.) +
(0.1) 16 282 kJ/kg (purch.) +
(0.1) 46 520 kJ/kg (Swan)

= 9 071 kJ/kg (3 900 Btu/lb)

Comparing (a) and (b) it can be seen that the energy saved by use of remanufacture is:
62 802 kJ/kg - 9 071 kJ/kg =
53 730.60 kJ/kg (23 100 Btu/lb).

With a minimum annual market of 1 818 436 L/g (400 000 gal/yr), this would result in an annual energy saving of $97\,482 \times 10^6$ kJ ($92\,400 \times 10^6$ Btu) and also eliminate the costs of transport of waste from the gas plant to Swan Hills. Transportation costs are not included in the estimates.

Section 5

Research and Development

Before deciding on a process scheme and arrangement, the founders of Canadian Chemical Reclaiming Ltd. studied alternative process systems which could obtain the recovery and purification of gas-treating chemicals.

A major concern from the beginning of the research was to avoid the possible decomposition of the chemical due to excessive temperature.

A study of the alternatives indicated that distillation under high vacuum was the most promising recovery method. A small bench-scale pilot plant was constructed in 1987 to confirm the process and to obtain more data as for the design of a prototype unit. The bench-scale plant was set up to operate on a batch basis with a vacuum of about 0.81 kPa (24 in. Hg); heat input from a bunsen burner; and condensation by heat exchange with water; all carried out in glass equipment. All of the operations of the bench-scale plant had the objective of limiting the maximum temperature in the process to no more than 205°C (400°F).

Market investigation among the major gas plant operators showed that the principal needs to reprocess chemicals would probably be found with DEA and with triethylene glycol. The chemicals that were tested in the bench-scale plant included DEA, ethylene glycol, and triethylene glycol. The bench-scale plant was operated for about a year and provided good confirmation that the concept was viable both physically and economically and a prototype design could be developed.

5.1 Prototype Design

The bench-scale pilot plant study had positive results and the decision was taken to design the full-scale prototype unit. In order to better serve the gas plant operators, the principal objective chosen was to design and construct a portable process unit which could be mounted and transported on a standard trailer. The dimensions of the unit and its processes, therefore, were constrained by the limitations and specifications of the Department of Transport [i.e., height ~ 4.9 m (~ 16 ft), width ~ 2.6 m (~ 8 ft 6 in) and length of ~ 12.8 m (~ 42 ft)]. Although the feasibility was confirmed in the final detailed design, many technical and operating features were dictated by the prescribed dimensions.

For the process design, the main goal was to heat and vaporize the waste chemical without exposing it to excessive temperatures or extended residence times at elevated temperatures. As has been noted, the chemicals selected for design purposes all have a tendency to degrade or decompose when exposed to temperatures at or about 205°C (400°F). Critical design steps such as the control of bulk fluid temperatures, flow velocities in high temperature sections, and residence times in high temperature zones of the process had to be identified and verified.

To raise the feedstream temperature to the desired level, incoming solutions were designed to mix directly with large volumes of heated, recycled solutions. Further, with the capacity of the portable unit restricted by the limiting dimensions of the trailer, recycling of treated solutions proved to be an essential design consideration in the control of residence times and flow velocities through the various stages of the process.

The design flow of the recycle was about 40 times the flow of the feed, and was only limited by the size of the tubes (7.6-cm (3-in) schedule 80 are used) and the power that was required by the pump that recirculates the

recycle material. This concept requires both an efficient mixer and a high capacity vapour-liquid separator.

A compact inline mixer has been designed for the plant. A vapour-liquid separator (V-1 Still) has also been designed with internals that optimize velocity and centrifugal force. The vaporized material is the recycled product and the liquid is the heavy waste materials in the feed solution (Figure 1).

The design of the still heater was somewhat unusual in that only convection heat transfer was provided. This ensured that the tubes receiving heat do not "see" the flame and thereby avoid possible hot spots. The heater has also been designed with 100% or more excess combustion air to ensure that the maximum flue gas temperature will not exceed 1093°C (2000°F) and the flow of flue gas can be controlled under different operating conditions.

Corrosion was another important factor considered in the design of the unit and selection of equipment. Amine units are well known as having corrosion problems, and certain impurities such as salt in glycol solutions can also cause serious corrosion. In addition, a major concern was the high cost that would certainly occur if high alloy content was required for piping and equipment. A detailed review was made of literature concerning corrosion and the standards of National Association of Corrosion Engineers (NACE). At the conclusion of these studies, unalloyed carbon steel was chosen with the use of full stress relief in cases where higher temperatures or concentrations of chemicals would be encountered. In addition, extra wall thickness was provided above that which would be needed for mechanical strength in cases where higher corrosion rates were expected to occur, such as in the tubes of the still heater. Cast iron was used for all pump impellers and mechanical seals were employed on all pumps.

5.2 Plant Description

Figure 2 is a flow diagram of the plant. The various components of the plant are described in the following.

Feed Preheat (E-1). The feed to the plant enters at atmospheric pressure and is preheated, in a Shell and Tube Heat Exchanger (E-1). The exchanger recovers heat energy from the recycled still vapours and transfers this energy to the influent solution.

Still Feed Preparation (M-1). The preheated feed is next mixed with heated recirculated still bottoms. Chemicals are also added at this step. This is a critical step in the system. The heating to still temperature must be completed quickly and precisely.

The gas-treating chemical is vaporized at this stage, permitting its separation, cooling, and further processing.

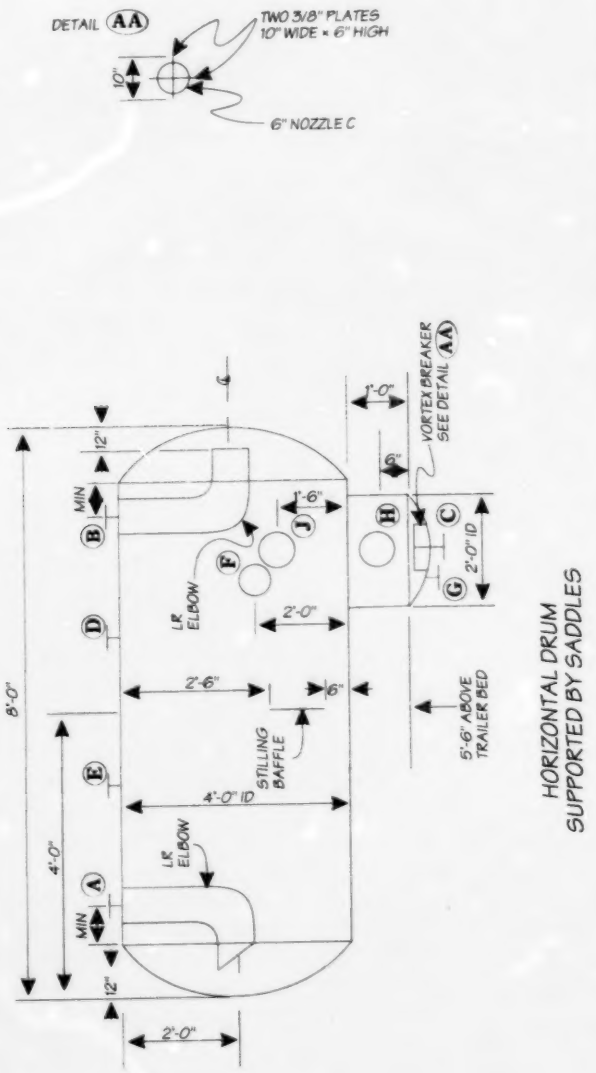
Still Separation (V-1). The mixture of feed and recirculated waste is fed to the Still V-1, where the gas-treating chemical is physically separated as a vapour from the heavy contaminants. The conditions of temperature and pressure are set to obtain the desired separation at the lowest practical temperature. A prescribed amount of treating chemical must remain in the waste bottoms to ensure the fluidity of the substance.

Still Heater (H-1). The still heater (H-1) is a natural gas fired heater which provides the energy to vaporize the chemical. A large volume of recirculated waste bottoms are heated over a short period of time to effect an incremental rise in temperature (Figure 3).

Still Vapour Exchange (E-1). The vapour from the Still are sent to a shell-and-tube heat exchanger, E-1, where they are cooled and partially condensed by exchange with the unit feed.

VESSEL DATA SHEET

Tag Number V-1 Item Name STILL



Design Conditions				
Interior	14.0	psig@	500	Deg F
Exterior	14.7	psig@	500	Deg F

Operating Conditions				
Top	2	psia@	360	Deg F
Bottom		psig@		Deg F

Material Specifications (NOTE: FULL STRESS RELIEF)			
Shell	C.S.	Heads	C.S.
Trays	C.S.	Internals	C.S.

Corrosion Allowance					
Shell	1/4"	Heads	1/4"	Internals	1/4"

CONNECTIONS

Mark	Size	Rating	Service
A	8"		VAPOR/LIQUID INLET FROM E-1
B	8"		VAPOR OUTLET TO E-1
C	6"		LIQUID OUTLET TO P-1
D	2"		RUPTURE DISK &RELIEF VALVE
E	20"		MANWAY
F	3/4"	6000#	LG-4
G	3/4"	6000#	LG-4
H	6"		LA HI/LO -1
J	6"		LAH-2
K			

Res. time Norm Level _____ Min
Level Control Range _____ Inches
Time @ Nrm Flow in Range _____ Min

FLOWS	
Inlet (lbs/hr)	Outlet (lbs/hr)

Figure 1 Vapour-Liquid Separator (Still V-1)

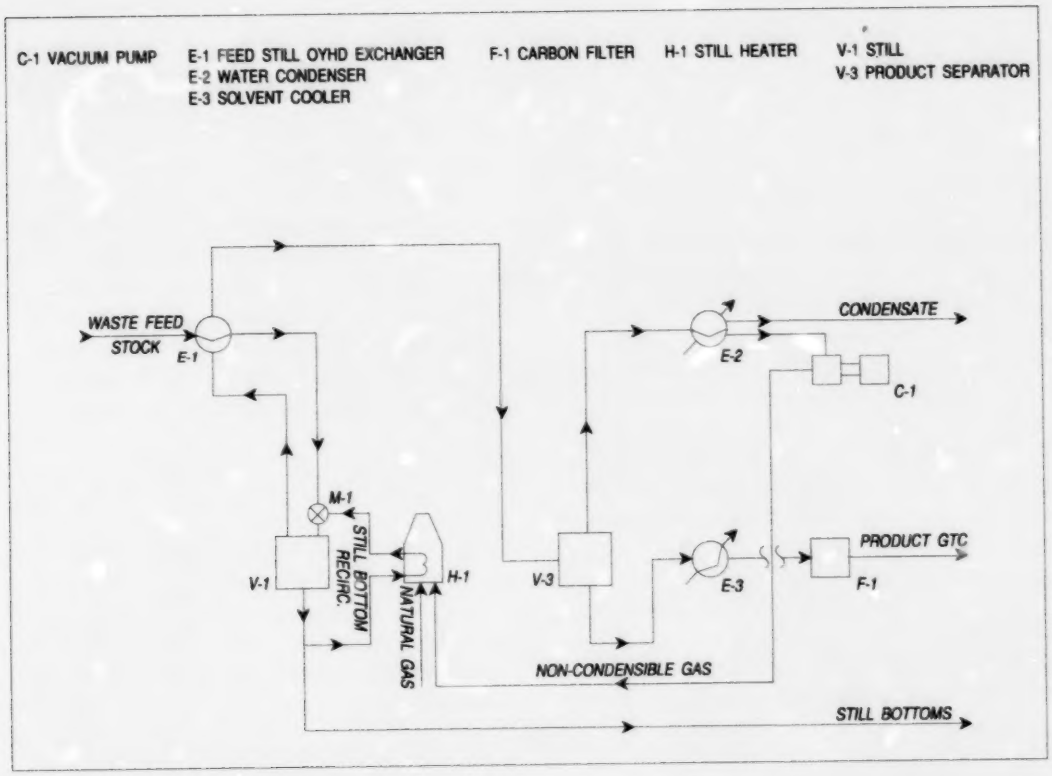


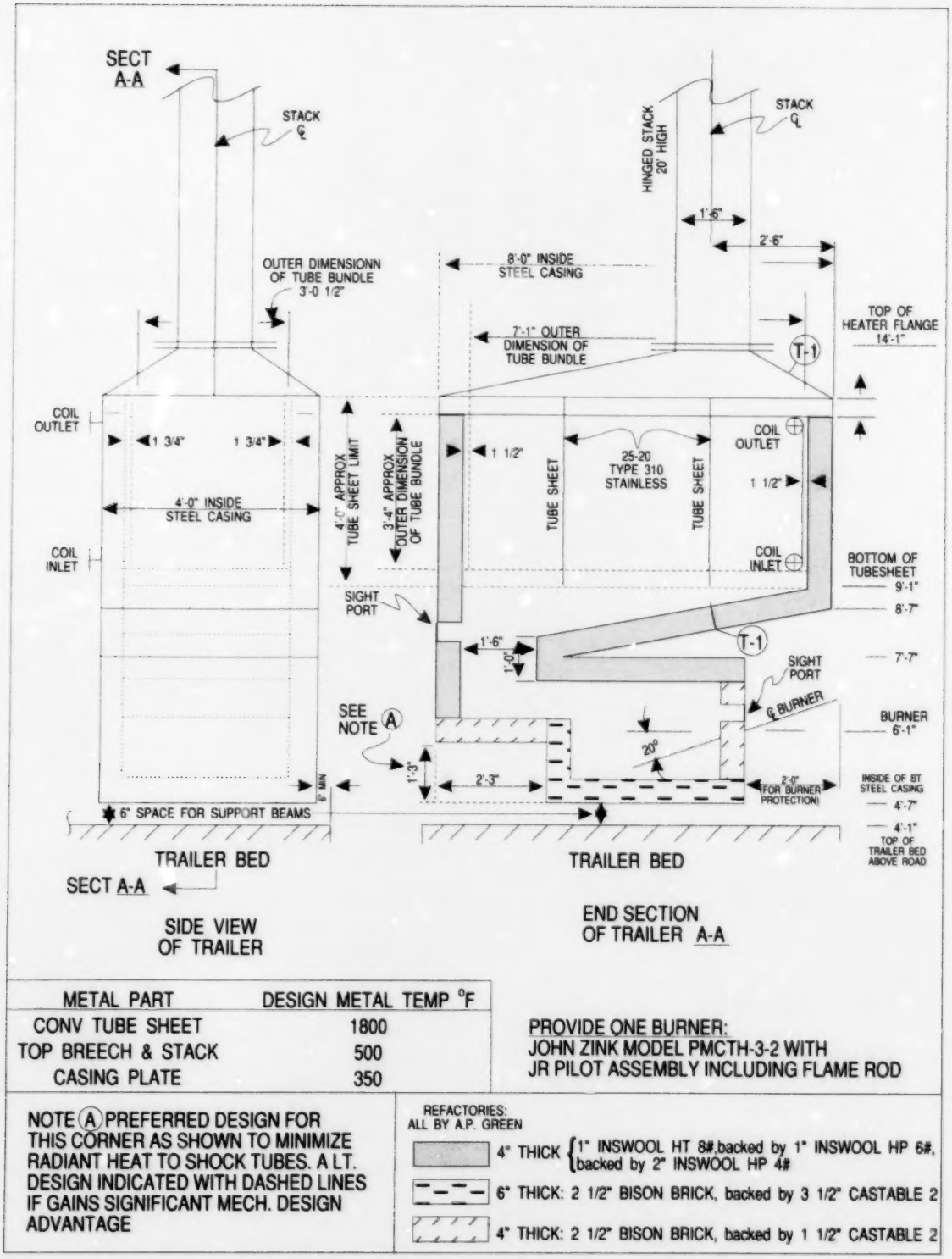
Figure 2 Flow Diagram

Product Separator (V-3). The product leaving Exchanger E-1 is a vapour-liquid mixture which flows to the Product Separator V-3 where the product chemical is separated as a liquid. This liquid is pumped through the Product Cooler E-3 and returned to the gas plant storage.

The vapour from the Product Separator flows to the Water Condenser E-2.

Water Condenser (E-2). The vapour flowing to the Water Condenser E-2 is water vapour with trace amounts of treating chemical and a small amount of air leakage and dissolved gases which may enter the unit. This vapour is cooled and condensed by exchange with cooling water in a Shell and Tube Heat Exchanger, E-2. It is then separated in the outlet channel into condensate liquid and non-condensable gases.

Vacuum Pump. The Vacuum Pump C-1 removes non-condensable gases from the bottom channel of the Water Condenser to maintain a high vacuum in the essential parts of the plant. The Vacuum Pump compresses these gases to slightly above atmospheric pressure after which they are sent to a special burner in the Still Heater. This method of disposal ensures that any residual hydrogen sulphide (H_2S) or other noxious gases are combusted prior to atmospheric release.



METAL PART	DESIGN METAL TEMP °F
CONV TUBE SHEET	1800
TOP BREECH & STACK	500
CASING PLATE	350

PROVIDE ONE BURNER:
JOHN ZINK MODEL PMCTH-3-2 WITH
JR PILOT ASSEMBLY INCLUDING FLAME ROD

NOTE (A) PREFERRED DESIGN FOR THIS CORNER AS SHOWN TO MINIMIZE RADIANT HEAT TO SHOCK TUBES. A LT. DESIGN INDICATED WITH DASHED LINES IF GAINS SIGNIFICANT MECH. DESIGN ADVANTAGE

REFACTORIES:
ALL BY A.P. GREEN

4" THICK {1" INSWOOL HT 8#,backed by 1" INSWOOL HP 6#, backed by 2" INSWOOL HP 4#

6" THICK: 2 1/2" BISON BRICK, backed by 3 1/2" CASTABLE 2

4" THICK: 2 1/2" BISON BRICK, backed by 1 1/2" CASTABLE 2

Figure 3 Natural Gas-fired Heater (H-1)

Section 6

History of Prototype Operations

During the past year, waste gas treating chemicals have been processed for a substantial number of gas plant operators. Chemicals have ranged from DEA to triethylene glycol, and sulfinol. The locations of the prototype testing, the chemical being remanufactured, its concentration, and the major contaminant removed are listed in Table 1.

6.1 Batch and Slipstream Operation

The original planning and design of the unit were based on the concept that the gas plant would be shut down and its inventory of chemical drained to a storage tank. This chemical would then be fed on a once through, or batch, basis to the reclaiming unit.

In a number of cases the gas plant has remained in operation during chemical processing.

6.2 Future Market Projection

The success of the prototype operation has led to an industry-wide awareness of the reclaiming process and its advantages. Market projections for the technology and its application to traditional gas treating chemicals are indeed encouraging.

Another potential source of revenue may be found in the processing of ethylene glycol currently used in automotive antifreeze and other heat transfer applications. Work is in progress to establish a collection system for materials of this nature.

Table 2 represents the results of a 12-month market projection, starting March 1, 1990.

Table 1 List of Operating Experience

Date	Location	Chemical Processed	I.G. Volume Processed	Major Contaminant
1988				
Aug.	Amoco Big Stone	Sulfinol 55% H_2O	14 000	Salt
Sept.	Chieftain Hight	Sulfinol 50% H_2O	15 000	Salt
Sept.	Canterra Hussar	EG 28% H_2O	12 000	Salt
Oct.	Canterra Ram River	DEA 63% H_2O	29 000	Heat-stable Salts
Oct.	Chevron Red Deer	EG 50% H_2O	16 000	Salt and Oil
Nov.	Union Carbide Red Deer	TEG 2% H_2O	7 019	H_2S
Nov.	Northwest Utilities	TEG	4 200	Oil and Salt
Nov.	SaskPower Red Deer	TEG 55% H_2O	6 500	Water
Dec.	Amoco Vulcan	Sulfinol 58% H_2O	14 000	HPM Oxazolidone
1989				
Jan.	Mobil Oil Harmattan	Sulfinol 54% H_2O	14 000	HPM Oxazolidone
Apr.	Amoco Vulcan	DEA 68% H_2O	17 000	Degradation Products
May	Mobil Oil Carson Creek	MDEA 50% H_2O	9 000	Salt
June	Petro-Canada Greencourt	DEA 65% H_2O	10 000	Salt
June	Amoco Wembly	DEA 70% H_2O	10 000	H_2O Removal
July	Harcross Red Deer	EG 50% H_2O	12 000	
July	Alt. Energy Red Deer	TEG 20% H_2O	8 100	Salt and Oil
Aug.	Amerada Min. Red Deer	TEG	5 500	Salt and Oil

Table 2 Market Survey for Chemicals

Chemical Group	Chemical	Amount L (gal.)
Amines		
	DEA	1 035 000 (230 000)
	Sulfinol	1 350 000 (300 000)
	MEA	315 000 (70 000)
	MDEA	450 000 (100 000)
Glycols		
	Ethylene Glycol-Heat Transfer Solutions	585 000 (130 000)
	Diethylene Glycol	90 000 (20 000)
	Triethylene Glycol	315 000 (70 000)
Total Projected Market Value		4 140 000 (920 000)

Section 7

Performance Test Results

Three performance test runs have been completed; one on DEA feed, one on ethylene glycol feed, and one on triethylene glycol feed. These tests were performed from June 6 through July 6, 1989; from May 7 through June 7, 1989; and from July 7 through August 7, 1989, respectively. Flows, temperatures, and pressures for these tests are tabulated in Table 3. Analytical results are presented in the Appendix.

The operating data presented in Table 3 represent average conditions over the time periods when the unit was in stable operation and operating at the design flow rate. It should be noted that the unit has been successfully operated at flow rates 20% higher than design rates.

In the first of the tests, processing DEA for Petro-Canada, the objective of the client was salt removal (NaCl). Starting with the feed solution which contained about 40 000 ppm NaCl, a product was delivered which contained only about 650 ppm or a removal of better than 98%. One requirement that governs the loss of chemical is that sufficient chemical be left in the waste bottoms to ensure the fluidity and pumpability of the waste. With waste bottoms containing about 55% by weight NaCl, it was necessary to allow a DEA loss of slightly over ten percent. The client was quite satisfied with these results.

In the second test, processing ethylene glycol for Harcross Chemical, the objective of the client was to remove water from the solution for recycle to the dehydration process. The feed solution contained about 49% water. A product was delivered with a water content of only 7%. In addition, over 75% of the chlorides and suspended solids were removed. The client was quite satisfied with the results.

In the third test, processing triethylene glycol for the Alberta Energy Corp., the objective of the client was to remove salt and oil. The feed solution contained about 500 ppm of salt, and about 5 ppm of oil. The product was delivered with 21 ppm salt and less than 1 ppm oil. In addition, the water content was reduced from 49% to 7.5%. The client was quite satisfied with these results.

7.1 Plant and Equipment Performance Evaluation

The operating results obtained indicate that the unit successfully reached design throughput capacities while removing target impurities to desired levels. Overall, it is clear that the design and operating objectives of the unit have been proven.

In the following discussion, individual items of equipment are examined to determine if each has successfully attained its operating function.

The **E-1, Feed - Still Overhead Exchanger** was deficient in its ability to transfer heat throughout the performance test program. During the performance tests it was calculated that the exchanger transferred only 65 to 75% of expected heat duty. More heat had to be added to the feed in the still heater to make up for the poor performance of the heat exchanger. The excess heat was balanced by increasing the cooling capacity of the water condenser.

A major part of the problem rests with the original design of the heat exchanger (E-1) and it is likely that more transfer surface should have been installed. When another operating unit is built it is planned to improve this design to provide more surface area. For the existing unit, the cost and downtime that

Table 3 Performance Test Operating Data

	Feed		
	DEA*	Ethylene Glycol**	Triethylene Glycol**
Concentration (Wt. %)	35%	50%	50%
Feed Flow, L/h	932	892	955
Prod. Flow, L/h	277	364	396
Cond. Flow, L/h	582	505	473
Waste Flow, L/h	73	23	86
Nat. Gas Flow, m^3/h	36.5	53.8	42.5
	Temperatures (°C)		
Feed to E-1	27	19	22
Feed from E-1	59	61	59
Waste from V-1	175	109	177
Waste from F-1	182	121	187
Prod. V to E-1	166	104	166
Prod. from E-1	62	87	69
Vapour to E-2	62	87	69
Liquid from E-2	27	29	27
Prod. Liq. to Client	62	54	52
Water to E-2	21	22	21
Water from E-2	30	31	29
H-1 Firebox	954	1038	1010
H-1 Stack	316	354	343
	Pressures (kPa Absolute)		
Feed to E-1	51.2	50.2	50.2
Feed from E-1	18.7	16.6	18.7
Discharge P-3	203	180	173
Prod. V to E-1	18.0	15.9	17.6
Prod. L from P-2	263	270	249
Vapour to E-2	14.2	14.5	14.2
Vapour from E-2	12.5	13.2	13.2
Discharge C-1	101	101	101
Prod. L to Client	166	201	194
Discharge P-4	111	104	118
Discharge P-5	194	187	194
Nat. Gas to Burner	132	145	145

Dates in 1989

* June 6 to July 6

** May 7 to June 7

*** July 7 to August 7

would be necessary to replace the exchanger rule out such a change, especially since the overall unit performance has been satisfactory.

The **E-2, Water Condenser** has performed satisfactorily. Only when feedstocks with high water content (>30%) are being processed does the water condenser become the factor which limits the capacity of the overall system.

The water going to the condenser has been acidified from time to time in order to avoid and/or remove scale deposition on the shell (water) side of the exchanger.

Difficulties have been encountered with the separation of vapour and liquid on the outlet of the condenser. The design provides for the use of the bottom channel head with baffle to obtain the desired separation. A future design could benefit by enlargement and/or lengthening of this channel to obtain better separation.

E-3, Solvent Cooler. No difficulties have been encountered with the E-3 and, in fact, it is often possible to operate successfully without using this part of the plant.

The **E-4, Water Cooler** (a small cooling tower) is one of the key equipment items in the plant. There have been no operating difficulties with this Water Cooler and in fact excellent service and performance have been obtained.

The **V-1, Still** serves to separate the vapour from the liquid entering the vessel. This separator has been operated at above design throughputs and has provided satisfactory separations and product quality.

The operation of the **V-3, Product Separator** is less critical than that of V-1. Fully satisfactory performance has been obtained from this separator at throughput rates above design.

The **H-1, Still Heater** has provided the necessary heat input to the process and, due to the deficiency of heat input from E-1, has provided higher than design heat input. From the standpoint of heat transfer and pressure drop, this heater has been entirely satisfactory.

The function of the **M-1, Mixer** is to heat the feed by direct mixing with a stream of heated recycle from the heater H-1. All available indications are that this mixer is performing as expected and doing a satisfactory job.

C-1 Vacuum Pump. The ability to maintain high vacuum in the plant is determined by the quality of the joints in the plant and by the capacity and reliability of the vacuum pump. Outstanding service has been obtained from the vacuum pump both in performance and in reliability.

The **P-1, Gauge Pump** is not normally used. The feedstock is usually obtained through direct piping to the client source. The vacuum in the system provides the pressure drop to create flow.

P-2, Product Solvent Pump. Some trouble was experienced in the initial operations with inadequate net positive suction head (NPSH) for the product solvent pump. The original impeller was replaced and the driving motor was replaced with a higher speed motor. After these changes there has been no further problem.

The **P-4, Condensate Pump** has performed satisfactorily and to all design requirements.

The **P-5, Cooling Water Pump** has also performed quite satisfactorily.

7.2 Plant Operating Problems

There have been two major operating problems and both concern the system which circulates waste bottoms from the Still (V-1) back through the heater (H-1) to the mixer (M-1).

The first problem was caused by the mechanical seal on the recirculation pump. The seal never functioned correctly and

caused numerous operating problems. Air leakage was experienced through the seal, causing pump cavitation and reducing the capacity of the pump and the system. It was later learned that the supplier had overlooked the fact that the pump suction would be under a high vacuum and that the selected seal was inappropriate for this purpose.

It was also discovered that the o-rings which were essential to the seal operation were not resistant to the chemicals being pumped. Following experimentation with various materials, it was resolved that the o-rings had to be replaced on each and every shutdown. Even with the most resistant material, Kal-Rez, satisfactory operation and performance could not be obtained.

After much lost production and out-of-pocket expense, it was decided to replace the entire seal with a new, double-seal arrangement. Since the installation of this much more expensive and complicated system, no further operating problems have been observed with this pump system.

The second problem was caused by the combination of a low flow rate to the gas-fired heater and an excessive firing rate to the heater. These incidents culminated in a perforation and failure of the tube bundle in the heater.

The low flow rate was at least partly caused by the seal problems previously mentioned. The operators throttled the discharge pump in order to minimize cavitation, resulting in low flow to the heater. In an attempt to maintain capacity of the unit, the operators then raised the firing rate to the heater. The combination of low flow and high firing caused hotspots to occur near the outlet of the heater and resulted in highly accelerated corrosion.

A larger impeller has been installed in the waste-circulating pump to ensure that the flow through the heater is more than adequate. In addition, a thermocouple has been installed in the fired heater to provide continuous temperature readings. A newly fabricated tube bundle has also been installed in the heater. No further operating problems have occurred to date.

7.3 *Environmental Impact Measurements*

7.3.1 Gaseous Emissions

The only fuel to the unit is natural gas; therefore, it was not expected to encounter emissions that would have a negative effect on air quality. The *Alberta Clean Air Act* requirements for ambient air are listed in Table 4, along with the results of emission tests performed on the plant's stack gases. It should be noted that the sampling of emissions was performed on a spot basis. As the unit is portable, it will be operated under the clean air permit of the client. The emissions testing program was undertaken to establish a nominal base level of emissions for the unit.

The tests for metals in the stack emissions revealed concentrations of less than 1.0 mg/m^3 for all metals with the exception of calcium, potassium, sodium, and silicon. One test indicated a zinc concentration of 2.1 mg/m^3.

7.3.2 Liquid Emissions

There are three liquid streams produced in the plant. The product, or purified gas-treating chemical, is returned to the plant for re-use in the gas-treating process.

The bottoms from the still are also returned to the client, placed in drums, and sent to Swan Hills. This waste material contains the vast majority of contaminants such as: salt, asphatenes, heat stable salts, and degradation products, including oxazolidones and piperazines. The volume of waste varies with each feedstock but generally is in the range of five to ten percent of the feed to the unit.

The condensate from the Water Condenser E-2 may contain some impurities. In cases where most of the water is being removed

Table 4 Requirements for Ambient Air*

	Alberta Maximum Level (mg/m^3 - 24h)	Stack Analysis (spot basis) (mg/m^3)
SO_2	150	2.2
NO_2	200	94
Particulate	100	19
Phenolics		0.05
Chlorinated Hydrocarbons		0.099

* *Alberta Clean Air Act*

from the solvent, and where the solvent has a low boiling point (such as ethylene glycol), this stream may have a significant content of the gas-treating chemical. This will in turn result in a high chemical oxygen demand (COD), which can reach levels as high as 200 000 ppm.

The waste condensate may contain, in addition to a small amount of solvent, small quantities of light hydrocarbons, and even smaller quantities of dissolved hydrogen sulphide and carbon dioxide.

Clients dispose of the condensate in various approved manners. The stream is not released to the environment.

7.4 Future Work and Development

There are two major areas which have been programmed for future work. The most important of these is the addition of a refluxed packed section in the top part of V-3, Product Separator. It has been determined that the unit is not as efficient as desired when removing high concentrations of water from gas-treating chemicals. Sometimes the carryover of gas-treating chemical to the waste condensate is higher than is desirable.

A preliminary design of the packed section has been prepared. During the winter, it is planned to shut the unit down and add the packed section complete with the necessary piping and valves to the top of the product separator.

The second item for future work is the Feed - Still Overhead Exchanger. It is planned to redesign this exchanger and thereby obtain more efficient recovery of heat. This will allow improved thermal efficiency of the unit.

Section 8

Conclusions

During the first year of operation a wide range of gas treating chemicals including DEA, EG, Sulfinol, MDEA, DEG, and TEG were successfully remanufactured at the plant. There is good evidence that the technology is a proven process that allow industry to reclaim its gas-treating chemical wastes.

Experience has shown that the requirements of the client often vary from the original design of the reclaimer. The objective to recover at least 90 to 95% of the solvent in the feed as remanufactured product has been met on many occasions during 1989. For various reasons, this objective was not met on any of the performance tests during this project. In the test on DEA, the objective of the client was to remove salt. To do so and to maintain the waste bottoms in a fluid state, it was necessary to allow more than ten percent of the DEA to remain in the waste bottoms. While this did not comply with the design objective, the client was completely satisfied with the results.

The three performance tests have demonstrated the objective of high removal of contaminants. In each case, removal of the contaminant specified by the client was successfully achieved. These removals ranged from 85% (in the case of water removal) to 98% in the case of salt removal.

In general, the remanufactured chemical has been returned to the client with uniform satisfaction. In a few cases, the remanufactured chemical has been marketed to a company, other than the client company. It has been clearly demonstrated that the remanufactured product is "marketable".

In each of the test runs, the waste products from the plant have been successfully disposed of in a manner considered acceptable by the Alberta Ministry of Environment.

References

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Appendix

Performance Test Laboratory Analyses

The analytical results from Core Laboratories and from Western Research for the performance tests are transmitted separately as attachments. These analytical results appear in unedited form.

SUMMARY OF EMISSION TEST RESULTS

CANADIAN CHEMICAL RECLAIMING
ALBERTA

JUNE/JULY 1989
FILE 5418/89-1

TEST DATE	June 6	July 5	July 7
LOCATION	at Greencourt	at Red Deer	at Red Deer
TEST START	09:07(DEA)	10:32(Eth.) (Glycol)	12:18(Trieth.) (Glycol)
TEST END	12:20	12:36	14:31
STACK GAS COMPOSITION		Mole Percents	
OXYGEN	13.83	12.22	15.08
CARBON DIOXIDE	3.34	3.12	2.86
CARBON MONOXIDE	<.0001	0.0026	0.0019
SULPHUR DIOXIDE	<.0001	<.0001	<.0001
WATER	7.04	7.64	6.35
NITROGEN	75.79	77.02	75.71
	MG/M3 @ 25.0°C and 760 mm Hg (dry basis)		
PARTICULATES			
Front Half	32.7	2.5	2.2
Back Half	7.9	5.3	6.5
Total Train	40.5	7.8	8.7
CHLORINATED HYDROCARBONS	0.020	0.148	0.128
PHENOLS	0.045	<.086	<.074
OXIDES OF NITORGEN AS NO_2	87.0	122.5	71.2
METALS			
Aluminum	0.165	ND	ND
Boron	0.990	0.017	0.018
Barium	0.038	0.011	ND
Calcium	2.246	0.242	ND
Chromiun	0.019	0.030	ND
Iron	0.337	0.014	0.071
Potassium	ND	ND	1.215
Molybdenum	0.057	ND	ND
Sodium	23.232	6.106	ND
Nickel	0.038	0.097	ND
Phosphorous	ND	0.278	0.106
Silicon	19.537	0.069	ND
Vanadium	ND	0.055	ND
Zinc	0.038	2.137	ND
TOTAL	46.697	9.056	1.410

Metals analyzed for but not detected include silver, arsenic, cadmium, copper, magnesium, manganese, lead, selenium, tin and cobalt.

ND - not detected

89/08/02
46-1888J

WESTERN RESEARCH, Calgary, Alberta

CORE LABORATORIES

COMPANY Canadian Chemical Reclaiming Ltd.

PAGE 1
FILE 52141-89-1276
DATE 1989 07 07

Date sampled: 1989 06 07
Date Received: 1989 06 22
Date Analyzed: 1989 07 07

Sampled From: DEA Unit Feed #1

Diethanolamine Analysis

		Mass Fraction
1.	Amine Concentration (by Alkalinity) as Diethanolamine	0.369
2.	Water Concentration (by Karl Fischer)	0.634
3.	Gas Chromatography	
	a. MEA	<0.001
	b. DEA	0.366
	c. TEA	<0.003
	d. AEEA (aminoethylethanolamine)	<0.003
	e. HEP (N, N-Di-(2-hydroxyethyl) piperazine)	<0.003
	f. HEOZD (3-(2-hydroxyethyl) oxazolidone-2	<0.003
4.	Total Nitrogen (Kjeldahl) as Diethanolamine	0.390
5.	Heat Stable Salts (expressed as DEA)	0.056
6.	Total Apparent Thiosulphate Content	<0.0005
7.	Chloride	23062 mg/kg
8.	Chloride Calculated as Sodium Chloride	38025 mg/kg
	a. Total Sulphides 5 gr/USG	90 mg/L
10.	Hydrocarbons	48 mg/L
11.	Total Suspended Solids	28 mg/L

CORE LABORATORIES

COMPANY Canadian Chemical Reclaiming Ltd.

FILE 52141-89-1276
DATE 1989 07 07

Date Sampled: 1989 06 07
Date Received: 1989 06 22
Date Analyzed: 1989 07 07

Sampled From: DEA Unit Feed #2

Diethanolamine Analysis

		Mass Fraction
1.	Amine Concentration (by Alkalinity) as Diethanolamine	0.326
2.	Water Concentration (by Karl Fischer)	0.658
3.	Gas Chromatography	
	a. MEA	<0.001
	b. DEA	0.342
	c. TEA	<0.003
	d. AEEA (aminoethylethanolamine)	<0.001
	e. HEP (N, N-Di-(2-hydroxyethyl) piperazine)	<0.003
	f. HEOZD (3-(2-hydroxyethyl) oxazolidone-2	<0.003
4.	Total Nitrogen (Kjeldahl) as Diethanolamine	0.330
5.	Heat Stable Salts (expressed as DEA)	0.069
6.	Total Apparent Thiosulphate Content	<0.0005
7.	Chloride	24700 mg/kg
8.	Chloride Calculated as Sodium Chloride	40725 mg/kg
9.	Total Sulphides 10 gr/USG	163 mg/L
10.	Hydrocarbons	<1 mg/L
11.	Total Suspended Solids	10 mg/L

CORE LABORATORIES

COMPANY Canadian Chemical Reclaiming Ltd.

FILE 52141-89-1276
DATE 1989 07 07

Date Sampled: 1989 06 07
Date Received: 1989 06 22
Date Analyzed: 1989 07 07

Sampled From: DEA Unit Feed #3

Diethanolamine Analysis

			Mass Fraction
1.	Amine Concentration (by Alkalinity) as Diethanolamine		0.324
2.	Water Concentration (by Karl Fischer)		0.645
3.	Gas Chromatography		
	a. MEA		<0.001
	b. DEA		0.353
	c. TEA		<0.003
	d. Ethylene Glycol		0.002
	e. AEEA (aminoethylethanolamine)		<0.001
	f. HEP (N, N-Di-(2-hydroxyethyl) piperazine)		<0.003
	g. HEOZD (3-(2-hydroxyethyl) oxazolidone-2		<0.003
4.	Total Nitrogen (Kjeldahl) as Diethanolamine		0.322
5.	Heat Stable Salts (expressed as DEA)		0.073
6.	Total Apparent Thiosulphate Content		<0.0005
7.	Chloride	24671 mg/kg	
8.	Sodium Chloride	40678 mg/kg	
9.	Total Sulphides 9 gr/USG	145 mg/L	
10.	Hydrocarbons	<1 mg/L	
11.	Total Suspended Solids	12 mg/L	

COMPANY Canadian Chemical Reclaiming Ltd.

Date Sampled: 1989 06 07
Date Received: 1989 06 22
Date Analyzed: 1989 07 07

Location: DEA Unit Feed #1

Trace Metals Analysis

Element	Unit Feed #1 mg/kg	Unit Feed #2 mg/kg	Unit Feed #3 mg/kg
Aluminum	<0.5	<0.5	<0.5
Antimony	0.5	<0.5	0.5
Arsenic	1.5	0.5	0.4
Barium	0.3	0.3	0.4
Beryllium	<0.05	<0.05	<0.05
Boron	18	22	22
Cadmium	<0.05	<0.05	<0.05
Calcium	3	3	3
Chromium	<0.1	<0.1	<0.1
Cobalt	<0.3	<0.3	<0.3
Copper	<0.1	<0.1	<0.1
Iron	10	6	5
Lead	<0.5	<0.5	<0.5
Magnesium	2.0	2.1	2.3
Maganese	0.2	0.2	0.2
Molybdenum	<0.5	<0.5	<0.5
Nickel	<0.4	<0.4	<0.4
Phosphorous	0.6	0.7	<0.6
Potassium	430	430	440
Selenium	1.7	1.4	1.5
Silver	<0.1	<0.1	<0.1
Sodium	15270	15000	16830
Strontium	0.5	0.5	0.5
Thallium	<1.0	<1.0	<1.0
Tin	1.1	3.5	2.8
Titanium	0.1	0.2	<0.1
Vanadium	<0.5	<0.5	<0.5
Zinc	0.8	2.7	7.8

CORE LABORATORIES

COMPANY Canadian Chemical Reclaiming Ltd.

FILE 52141-89-1276
DATE 1989 07 07

Date Sampled: 1989 06 07
Date Received: 1989 06 22
Date Analyzed: 1989 07 07

Sampled From: DEA Product #1

Diethanolamine Analysis

		Mass Fraction
1.	Amine Concentration (by Alkalinity) as Diethanolamine	0.919
2.	Water Concentration (by Karl Fischer)	0.051
3.	Gas Chromatography	
	a. MEA	<0.001
	b. DEA	0.944
	c. TEA	<0.003
	d. AEEA (aminoethylethanolamine)	<0.001
	e. HEP (N, N-Di-(2-hydroxyethyl) piperazine)	0.005
	f. HEOZD (3-(2-hydroxyethyl) oxazolidone-2	<0.003
4.	Total Nitrogen (Kjeldahl) as Diethanolamine	0.915
5.	Heat Stable Salts (expressed as DEA)	0.003
6.	Total Apparent Thiosulphate Content	<0.0005

7.	Chloride		400 mg/kg
8.	Chloride Calculated as Sodium Chloride		659 mg/kg
9.	Total Sulphides	13 gr/USG	217 mg/L
10.	Hydrocarbons		55 mg/L
13.	Total Suspended Solids		340 mg/L

CORE LABORATORIES

COMPANY Canadian Chemical Reclaiming Ltd.

FILE 52141-89-1276
DATE 1989 07 07

Date Sampled: 1989 06 07
Date Received: 1989 06 22
Date Analyzed: 1989 07 07

Sampled From: DEA Product #2

Diethanolamine Analysis

			Mass Fraction
1.	Amine Concentration (by Alkalinity) as Diethanolamine		0.961
2.	Water Concentration (by Karl Fischer)		0.039
3.	Gas Chromatography		
	a. MEA		<0.001
	b. DEA		0.958
	c. TEA		<0.003
	d. AEEA (aminoethylethanolamine)		<0.001
	e. HEP (N, N-Di-(2-hydroxyethyl) piperazine)		0.003
	f. HEOZD (3-(2-hydroxyethyl) oxazolidone-2		<0.003
4.	Total Nitrogen (Kjeldahl) as Diethanolamine		0.930
5.	Heat Stable Salts		0.002
6.	Total Apparent Thiosulphate Content		<0.0005
7.	Chloride	326 mg/kg	
8.	Chloride Calculated as Sodium Chloride	538 mg/kg	
8.	Total Sulphides 17 gr/USG	289 mg/L	
9.	Hydrocarbons	5 mg/L	
10.	Total Suspended Solids	256 mg/L	

CORE LABORATORIES

COMPANY Canadian Chemical Reclaiming Ltd.

FILE 52141-89-1276
DATE 1989 07 07

Date Sampled: 1989 06 07
Date Received: 1989 06 22
Date Analyzed: 1989 07 07

Sampled From: DEA Product #3

Diethanolamine Analysis

		Mass Fraction
1.	Amine Concentration (by Alkalinity) as Diethanolamine	0.901
2.	Water Concentration (by Karl Fischer)	0.067
3.	Gas Chromatography	
	a. MEA	<0.001
	b. DEA	0.928
	c. TEA	<0.003
	d. AEEA (aminoethylethanolamine)	<0.001
	e. HEP (N, N-Di-(2-hydroxyethyl) piperazine)	0.005
	f. HEOZD (3-(2-hydroxyethyl) oxazolidone-2	0.003
4.	Total Nitrogen (Kjeldahl) as Diethanolamine	0.900
5.	Heat Stable Salts (expressed as DEA)	0.117
6.	Total Apparent Thiosulphate Content	<0.0005
7.	Chloride	398 mg/kg
8.	Chloride Calculated as Sodium Chloride	656 mg/kg
9.	Total Sulphides 20 gr/USG	686 mg/L
10.	Hydrocarbons	7 mg/L
11.	Total Suspended Solids	260 mg/L

CORE LABORATORIES

COMPANY Canadian Chemical Reclaiming Ltd.

FILE 52141-89-1276
DATE 1989 07 07

Date Sampled: 1989 06 07
Date Received: 1989 06 22
Date Analysed: 1989 07 07

Trace Metals Analysis

	Composite Sample DEA Product (mg/kg)
Aluminum	<1
Arsenic	2
Barium	<0.2
Beryllium	<0.1
Boron	9
Cadmium	<0.1
Calcium	3.4
Chromium	<0.2
Cobalt	<0.6
Copper	<0.2
Iron	70
Lead	<1
Magnesium	0.7
Manganese	0.5
Molybdenum	<1
Nickel	1.0
Phosphorous	<1.2
Potassium	23
Selenium	3
Silicon	1
Silver	<0.2
Sodium	100
Thallium	<2
Tin	4
Titanium	<0.2
Vanadium	<1
Zinc	<0.2

CORE LABORATORIES

COMPANY Canadian Chemical Reclaiming Ltd.

FILE 52141-89-1276
DATE 1989 07 07

Date Sampled: 1989 06 07
Date Received: 1989 06 22
Date Analyzed: 1989 07 07

Sampled From: DEA Unit Waste (Bottoms) #1

Diethanolamine Analysis

		Mass Fraction
1.	Water Concentration (by Karl Fischer)	0.010
2.	Gas Chromatography	
	a. MEA	<0.001
	b. DEA	0.955
	c. TEA	<0.003
	d. AEEA (aminoethylethanolamine)	<0.001
	e. HEP (N, N-Di-(2-hydroxyethyl) piperazine)	0.035
	f. HEOZD (3-(2-hydroxyethyl) oxazolidone-2	<0.003
3.	Chloride	36.92%
4.	Chloride calculated as Sodium Chloride	60.87%
4.	Total Sulphides	53 mg/L
5.	Hydrocarbon	2 mg/L

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COMPANY Canadian Chemical Reclaiming Ltd.

FILE 52141-89-1276
DATE 1989 07 07

Date Sampled: 1989 06 07
Date Received: 1989 06 22
Date Analyzed: 1989 07 07

Sampled From: DEA Unit Waste (Bottoms) #2

Diethanolamine Analysis

		Mass Fraction
1.	Water Concentration (by Karl Fischer)	0.006
2.	Gas Chromatography	
	a. MEA	<0.001
	b. DEA	0.955
	c. TEA	<0.003
	d. AEEA (aminoethylethanolamine)	<0.001
	e. HEP (N, N-Di-(2-hydroxyethyl) piperazine)	0.039
	f. HEOZD (3-(2-hydroxyethyl) oxazolidone-2	<0.003
3.	Chloride	35.88%
4.	Chloride Calculated as Sodium Chloride	59.16%
5.	Total Sulphides	58 mg/L

CORE LABORATORIES

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Date Sampled: 1989 06 07
Date Received: 1989 06 22
Date Analyzed: 1989 07 07

Sampled From: DEA Unit Waste (Bottoms) #3

Diethanolamine Analysis

		Mass Fraction
1.	Water Concentration (by Karl Fischer)	0.006
2.	Gas Chromatography	
	a. MEA	<0.001
	b. DEA	0.960
	c. TEA	<0.003
	d. AEEA (aminoethylethanolamine)	<0.001
	e. HEP (N, N-Di-(2-hydroxyethyl) piperazine)	0.034
	f. HEOZD (3-(2-hydroxyethyl) oxazolidone-2	<0.003
3.	Chloride	29.7%
4.	Chloride Calculated as Sodium Chloride	49.0%
5.	Total Sulphides	48 mg/L

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Date Sampled: 1989 06 07
Date Received: 1989 06 22
Date Analyzed: 1989 07 07

Trace Metals Analysis of Diethanolamine

	Unit Waste Bottoms Sample #1 (mg/kg)	Unit Waste Bottoms Sample #2 (mg/kg)	Unit Waste Bottoms Sample #3 (mg/kg)
Aluminum	68	<1	<1
Arsenic	20	<1	5.3
Barium	31	13	13
Beryllium	<0.1	<0.1	<0.1
Boron	220	370	320
Cadmium	12	6.3	3.1
Calcium	410	450	760
Chromium	36	11	25
Cobalt	12	<0.6	<0.6
Copper	22	18	30
Iron	2900	3700	4600
Lead	<1	<1	<1
Magnesium	39	26	130
Manganese	16	17	17
Molybdenum	<1	3.2	<1
Nickel	12	31	55
Phosphorous	<1.2	100	<1.2
Potassium	5700	6300	5400
Selenium	190	94	49
Silicon	160	110	97
Silver	38	8.9	12
Sodium	242000	232000	184000
Thallium	<2	<2	83
Tin	71	<1	54
Titanium	14	19	27
Vanadium	7.1	<1	7.6
Zinc	10	46	28

COMPANY Canadian Chemical Reclaiming Ltd.

Date Sampled: 1989 06 07
Date Received: 1989 06 22
Date Analyzed: 1989 07 07

Sample: Condensate (Waste Water)

Gas Chromatography Analysis

	Condensate Waste Water Sample #1 (mass frac.)	Condensate Waste Water Sample #2 (mass frac.)	Condensate Waste Water Sample #3 (mass frac.)
Monoethanolamine (MEA)	<0.001	<0.001	<0.001
Diethanolamine (DEA)	<0.001	<0.001	<0.001
Triethanolamine (TEA)	<0.003	<0.003	<0.003
Aminoethylethanolamine (AEEA)	<0.001	<0.001	<0.001
N, N-Di-(2 hydroxyethyl Piperazine) (HEP)	<0.003	<0.003	<0.003
3-(2 hydroxyethyl) Oxazolidone-2 (HEOZD)	<0.003	<0.003	<0.003

Chemical Analysis

	Sample #1	Sample #2	Sample #3
Oil & Grease (mg/L)	44	83	<10
Total Suspended Solids (mg/L)	1	7	1
Chloride (mg/kg)	<0.5	<0.5	<0.5
Sodium Chloride (mg/kg)	<1	<1	<1
Total Sulphides (mg/L)	<0.05	<0.05	<0.1
Chemical Oxygen Demand (mg/L)	13750	18120	10940
Water Content (mass fraction)	0.99	0.99	0.99

CORE LABORATORIES

COMPANY Canadian Chemical Reclaiming Ltd.

FILE 52141-89-1276
DATE 1989 07 07

Date Sampled: 1989 06 07
Date Received: 1989 06 22
Date Analyzed: 1989 07 07

Trace Metals Analysis of Diethanolamine

	Composite DEA Unit Condensate mg/kg)
Aluminum	<0.5
Arsenic	<0.5
Barium	<0.2
Beryllium	<0.05
Boron	3
Cadmium	<0.5
Calcium	2.6
Chromium	<0.1
Cobalt	<0.3
Copper	<0.1
Iron	1.0
Lead	<0.5
Magnesium	0.6
Manganese	<0.1
Molybdenum	<0.5
Nickel	<0.4
Phosphorous	<0.6
Potassium	<10
Selenium	<0.5
Silicon	3
Silver	<0.1
Sodium	12
Thallium	<1
Tin	<0.5
Titanium	<0.1
Vanadium	<0.5
Zinc	0.1

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COMPANY Canadian Chemical Reclaiming Ltd.

FILE 52141-89-1276
DATE 1989 07 07

Date Sampled: 1989 06 07
Date Received: 1989 06 22
Date Analyzed: 1989 07 07

Sample: Composite Diethanolamine Waste Water Condensate

Analysis

Halogenated Compounds	<0.01 mg/L
Total Phenols	<0.05 mg/L

GC/ECD

Analyst: Phillip Oltmann, Ph.D.
George Hilderman

CORE LABORATORIES

COMPANY Canadian Chemical Reclaiming Ltd.

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Triethylene Glycol Analysis

		TEG Unit Feed #1	TEG Unit Feed #2	TEG Unit Feed #3
1.	Water (by Karl Fischer, %)	48.8	48.5	50.0
2.	Total Sulphides (mg/L)	<2	<2	<2
3.	Gas Chromatography			
	Water (%)	49.7	48.2	49.1
	Ethylene Glycol (%)	0.6	0.8	0.7
	Diethylene Glycol (%)	0.6	0.6	0.6
	Triethylene Glycol (%)	35.4	36.3	35.2
	Tetraethylene Glycol (%)	13.4	13.8	14.0
	Methanol (%)	0.3	0.3	0.4
	Total	100.0	100.0	100.0
4.	Chloride	290	300	310
5.	Calculated Sodium Chloride (mg/L)	478	495	511
6.	pH (1:1)	5.29	5.28	5.25
7.	Hydrocarbon (mg/L)	10	1	5
8.	Total Suspended Solids (mg/L)	124	122	185

COMPANY Canadian Chemical Reclaiming Ltd.

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Triethylene Glycol Analysis

		TEG Unit Product #1	TEC Unit Product #2	TEG Unit Product #3
1.	Water (by Karl Fischer, %)	9.2	3.7	9.5
2.	Total Sulphides (mg/L)	<2	<2	<2
3.	Gas Chromatography			
	Water (%)	8.5	3.4	9.1
	Ethylene Glycol (%)	1.8	0.9	1.0
	Diethylene Glycol (%)	1.7	1.3	1.3
	Triethylene Glycol (%)	76.5	79.7	64.9
	Tetraethylene Glycol (%)	11.5	14.7	23.7
	Methanol (%)	<0.1	<0.1	<0.1
	Total	100.0	100.0	100.0
4.	Chloride (mg/L)	21	20	21
5.	Calculated Sodium Chloride (mg/L)	35	33	35
6.	pH (1:1)	8.91	9.05	7.30
7.	Hydrocarbon (mg/L)	<1	<1	<1
8.	Total Suspended Solids (mg/L)	21	4.8	10.0

COMPANY Canadian Chemical Reclaiming Ltd.

FILE 52141-89-1276
DATE 1989 07 17

Triethylene Glycol Analysis

		TEG Unit Waste #1	TEG Unit Waste #2	TEG Unit Waste #3
1.	Water (by Karl Fischer, %)	4.1	0.4	0.4
2.	Total Sulphides (mg/L)	<2	<2	<2
3.	Gas Chromatography			
	Water (%)	3.9	-	-
	Ethylene Glycol (%)	0.2	<0.2	<0.2
	Diethylene Glycol (%)	0.4	0.2	0.2
	Triethylene Glycol (%)	59.9	42.0	54.2
	Tetraethylene Glycol (%)	35.6	57.8	45.6
	Methanol (%)	<0.1	<0.1	<0.1
	Total	100.0	100.0	100.0
4.	Chloride (mg/L)	1600	1900	4500
5.	Calculated Sodium Chloride (mg/L)	2638	3133	7419
6.	pH (1:1)	10.7	11.3	9.9
7.	Hydrocarbon (mg/L)	<1	<1	<1
8.	Total Suspended Solids (mg/L)	1232.0	2208	1300

COMPANY Canadian Chemical Reclaiming Ltd.

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Triethylene Glycol Analysis

		TEG Waste #1 Condensate	TEG Waste #2 Condensate	TEG Waste #3 Condensate
1.	Water (by Karl Fischer, %)	98.5	98.0	99.2
2.	Total Sulphides (mg/L)	<2	<2	<2
3.	Gas Chromatography			
	Water (%)	98.7	97.2	98.5
	Ethylene Glycol (%)	<0.2	0.8	0.2
	Diethylene Glycol (%)	<0.2	0.4	<0.2
	Triethylene Glycol (%)	0.7	1.0	0.7
	Tetraethylene Glycol (%)	<0.4	<0.4	<0.4
	Methanol (%)	0.6	0.6	0.6
	Total	100.0	100.0	100.0
4.	Chloride (mg/L)	9	14	20
5.	Calculated Sodium Chloride (mg/L)	14.8	23.1	33.0
6.	pH (Direct)	9.8	9.4	9.0
7.	Hydrocarbon	<1	<1	<1
8.	Total Suspended Solids (mg/L)	89.5	2.7	1.1
9.	Chemical Oxygen Demand (mg/L)	26150	35385	20000

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DATE 1989 07 17

Date Received: 1989 07 12
Date Analyzed: 1989 07 12

Trace Metals Analysis (mg/kg)

Element	TEG Unit Feed Composite	TEG Unit Product Composite	TEG Unit Waste Composite	TEG Waste Water Condensate Composite
Aluminum	<1	<1	2	<1
Arsenic	1	2	2	<1
Barium	<0.2	<0.2	0.6	<0.2
Beryllium	<0.1	<0.1	<0.1	<0.1
Boron	4	2	26	<1
Cadmium	<0.1	<0.1	<0.1	<0.1
Calcium	13	0.8	47	0.3
Chromium	<0.2	<0.2	<0.2	<0.2
Cobalt	<0.8	<0.8	<0.8	<0.8
Copper	<0.2	<0.2	0.3	<0.2
Iron	120	8.8	500	0.7
Lead	<1	<1	<1	<1
Magnesium	3.1	<0.2	12	<0.2
Manganese	5.0	0.3	18	<0.2
Mercury	<1	<1	<1	<1
Molybdenum	<1	<1	<1	<1
Nickel	<1	<1	<1	<1
Phosphorous	3	<2	54	<2
Potassium	380	26	1600	<25
Selenium	1	3	4	<1
Silicon	3	2	20	4
Silver	<0.2	<0.2	<0.2	<0.2
Sodium	250	57	2600	1
Thallium	<2	<2	<2	<2
Tin	3	9	8	<1
Titanium	<0.2	<0.2	<0.2	<0.2
Vanadium	<1	<1	<1	<1
Zinc	1.5	<0.2	6.4	<0.2
Strontium	0.7	<0.2	2.6	<0.2

CORE LABORATORIES

COMPANY Canadian Chemical Reclaiming Ltd.

FILE 52141-89-1276
DATE 1989 07 07

Date Sampled: 1989 06 07
Date Received: 1989 06 22
Date Analyzed: 1989 07 07

Sample: Composite Triethylene Glycol Waste Water Condensate

Analysis

Halogenated Compounds	<0.01 mg/L
Total Phenols	<0.05 mg/L

GC/ECD

Analyst: Phillip Oltmann, Ph.D.
George Hilderman

COMPANY Canadian Chemical Reclaiming Ltd. PAGE 22
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DATE 1989 07 17

Ethylene Glycol Analysis

		EG Unit Feed #1	EG Unit Feed #2	EG Unit Feed #3
1.	Water (by Karl Fischer, %)	49.5	49.6	48.7
2.	Total Sulphides (mg/L)	<2	<2	<2
3.	Gas Chromatography			
	Water (%)	49.0	49.0	48.0
	Ethylene Glycol (%)	48.3	48.0	49.3
	Diethylene Glycol (%)	<0.2	<0.2	0.2
	Triethylene Glycol (%)	<0.2	0.2	0.6
	Tetraethylene Glycol (%)	<0.4	<0.4	<0.4
	Methanol (%)	2.7	2.8	1.9
	Total	100.0	100.0	100.0
4.	Chloride (mg/L)	138	32	55
5.	Calculated Sodium Chloride (mg/L)	227.5	52.8	90.7
6.	pH (1:1)	8.6	8.5	8.6
7.	Hydrocarbon	<1	<1	5
8.	Total Suspended Solids (mg/L)	36.0	65.0	172.0

COMPANY Canadian Chemical Reclaiming Ltd.

FILE 52141-89-1276
DATE 1989 07 17

Ethylene Glycol Analysis

		EG Unit Product #1	EG Unit Product #2	EG Unit Product #3
1.	Water (by Karl Fischer, %)	10.8	6.5	4.0
2.	Total Sulphides (mg/L)	<2	<2	<2
3.	Gas Chromatography			
	Water (%)	10.3	6.1	3.7
	Ethylene Glycol (%)	89.3	93.9	96.1
	Diethylene Glycol (%)	<0.2	<0.2	<0.2
	Triethylene Glycol (%)	0.3	<0.2	0.2
	Tetraethylene Glycol (%)	<0.4	<0.4	<0.4
	Methanol (%)	0.1	<0.1	<0.1
	Total	100.0	100.0	100.0
4.	Chloride (mg/L)	<10	25	25
5.	Calculated Sodium Chloride (mg/L)	<16.0	41.2	41.2
6.	pH (1:1)	10.1	10.0	10.1
7.	Hydrocarbon (mg/L)	<1	<1	8
8.	Total Suspended Solids (mg/L)	1.8	6.0	41.0

CORE LABORATORIES

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Ethylene Glycol Analysis

		EG Unit Waste #1	EG Unit Waste #2	EG Unit Waste #3
1.	Water (by Karl Fischer, %)	1.4	23	1.4
2.	Total Sulphides (mg/L)	<2	<2	<2
3.	Gas Chromatography			
	Water (%)	1.1	24	1.3
	Ethylene Glycol (%)	92.3	85.1	84.9
	Diethylene Glycol (%)	1.5	2.3	2.4
	Triethylene Glycol (%)	5.1	10.2	11.4
	Tetraethylene Glycol (%)	<0.4	<0.4	<0.4
	Methanol (%)	<0.1	<0.1	<0.1
	Total	100.0	100.0	100.0
4.	Chloride (mg/L)	1216	1168	1593
5.	Calculated Sodium Chloride (mg/L)	2005	1926	2627
6.	pH (1:1)	11.3	10.9	11.2
7.	Hydrocarbon (mg/L)	<1	<1	<1
8.	Total Suspended Solids (mg/L)	678.0	280.0	750.0

CORE LABORATORIES

COMPANY Canadian Chemical Reclaiming Ltd.

FILE 52141-89-1276
DATE 1989 07 17

Ethylene Glycol Analysis

		EG Waste #1 Condensate	EG Waste #2 Condensate	EG Waste #3 Condensate
1.	Water (by Karl Fischer, %)	85.0	82.7	65.2
2.	Total Sulphides (mg/L)	<2	<2	<22.
3.	Gas Chromatography			
	Water (%)	85.2	81.6	64.5
	Ethylene Glycol (%)	9.8	13.0	33.7
	Diethylene Glycol (%)	<0.2	<0.2	<0.2
	Triethylene Glycol (%)	<0.2	<0.2	<0.2
	Tetraethylene Glycol (%)	<0.4	<0.4	<0.4
	Methanol (%)	5.0	5.4	1.7
	Total	100.0	100.0	100.0
4.	Chloride (mg/L)	9	10	9
5.	Calculated Sodium Chloride (mg/L)	14.8	16.5	14.8
6.	pH (Direct)	8.4	8.4	8.0
7.	Hydrocarbon (mg/L)	<1	<1	<1
8.	Total Suspended Solids (mg/L)	2.3	5.0	11.0
9.	Chemical Oxygen Demand (mg/L)	181530	281525	438445

CORE LABORATORIES

COMPANY Canadian Chemical Reclaiming Ltd.

FILE 52141-89-1276
DATE 1989 07 17

Date Received: 1989 07 12
Date Analyzed: 1989 07 12

Trace Metals Analysis (mg/kg)

Element	EG Feed Composite	EG Product Composite	EG Waste Bottoms Composite	EG Waste Water Condensate Composite
Aluminum	<1	<1	1	<1
Arsenic	<1	1	7	<1
Barium	<0.2	0.3	<0.4	0.2
Beryllium	<0.1	<0.1	<0.1	<0.1
Boron	120	8	800	2
Cadmium	<0.1	<0.1	<0.1	<0.1
Calcium	3	0.3	16	0.6
Chromium	<0.2	<0.2	0.2	<0.2
Cobalt	<0.8	<0.8	<0.8	<0.8
Copper	<0.2	<0.2	0.2	<0.2
Iron	11	8.8	100	0.6
Lead	<1	<1	<1	<1
Magnesium	0.7	<0.2	3.7	0.2
Manganese	<0.2	<0.2	1.2	<0.2
Mercury	<1	<1	<1	<1
Molybdenum	<1	<1	2	<1
Nickel	<1	<1	2	<1
Phosphorous	260	6	920	<2
Potassium	680	37	4400	<25
Selenium	1	3	2	<1
Silicon	5	1	42	<1
Silver	<0.2	<0.2	<0.2	<0.2
Sodium	380	43	4600	4
Thallium	<2	<2	<2	<2
Tin	3	6	7	2
Titanium	<0.2	<0.2	0.7	<0.2
Vanadium	<1	<1	1	<1
Zinc	<0.2	0.3	1.2	<0.2
Strontium	<0.2	<0.2	0.3	<0.2

COMPANY Canadian Chemical Reclaiming Ltd.

Date Sampled: 1989 06 07
Date Received: 1989 06 22
Date Analyzed: 1989 07 07

Sample: Composite Ethylene Glycol Waste Water Condensate

Analysis

Halogenated Compounds	<0.01 mg/L
Total Phenols	<0.05 mg/L

GC/ECD

Analyst: Phillip Oltmann, Ph.D.
George Hilderman